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METHODS FOR DETERMINATION OF ORGANIC PRESERVATIVES IN FOODSTUFFS

PART 3 SORBIC ACID AND ITS SALTS

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PART 3 SORBIC ACID AND ITS SALTS

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Indian Standard

METHODS FOR DETERMINATION OF ORGANIC PRESERVATIVES IN FOODSTUFFS

PART 3 SORBIC ACID AND ITS SALTS

0. FOREWORD

0.1 This Indian Standard (Part 3) was adopted by the Indian Standards Institution on 30 October 1986, after the draft finalized by the Food Hygiene Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 For protecting food from microbial deterioration, a number of methods, such as application of heat or cold, dehydration, fermentation, irradiation or addition of certain chemicals, are employed. Besides extending the period of use of a food, a chemical preservative should be safe for human consumption, should not impart undesirable organoleptic changes, be economical in use and be capable of being analysed. While the use of chemical preservative to be safe under conditions of use is governed by law, it is considered necessary to prescribe methods for their analysis. The use of these methods would not only ensure repeatable and reproducible results for their correct interpretation, but would also facilitate interlaboratory comparisons.

0.3 The *Prevention of Food Adulteration Act*, 1954 and the Rules framed thereunder allow the use of two classes of preservatives, class I and class II. Class I preservatives include common salt, sugar, dextrose, glucose (syrup), wood smoke, spices, vinegar or acetic acid, honey, etc. Class II preservatives include inorganic substances like sulphurous acid including salts thereof, nitrates of sodium or potassium, and nisin, and organic substances like benzoic acid including salts thereof, sorbic acid including its sodium, potassium and calcium salts, and sodium and calcium propionate.

0.4 This standard covering the determination of organic preservatives is being issued in three parts. This part (Part 3) covers the determination of sorbic acid and its salts in foodstuffs. Part 1 covers the determination of benzoic acid and its salts and Part 2 covers the determination of propionic acid and its salts.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 3) prescribes the methods for determination of sorbic acid used as preservatives in foodstuffs.

2. QUALITY OF REAGENTS

2.1 Pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — Pure chemicals shall mean chemical that do not contain impurities which affect the results of analysis.

3. GENERAL

3.1 This standard specifies two methods for the determination of sorbic acid, viz colorimetric and spectrophotometric methods. Colorimetric method is applicable to cheese and flour confectionery products only where as the spectrophotometric method is applicable to fresh dairy products like cheese, sour cream, yoghurt and flour confectionery products.

4. COLORIMETRIC METHOD

4.1 Reagents

4.1.1 *Sulphuric Acid*—0·3 and 2N.

4.1.2 *Potassium Dichromate Solution* — Dissolve 147 mg. potassium dichromate in distilled water and dilute to 100 ml.

4.1.3 *Thiobarbituric Acid Solution (0·5 percent)*—Dissolve 250 mg thiobarbituric acid in 5 ml 0·5N sodium hydroxide solution in a 50 ml volumetric flask by swirling under hot water. Add 20 ml distilled water, neutralise with 3 ml 1N HCl and dilute to volume with distilled water. This solution should be prepared freshly before experimentation.

4.1.4 *Crystalline Magnesium Sulphate* — $MgSO_4$, $7H_2O$.

4.1.5 *Standard Sorbic Acid Solution* — Accurately weigh 134 mg potassium sorbate (equivalent to 100 mg sorbic acid) and dilute to

*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

1 litre with distilled water. 1 ml of solution corresponds to 0·1 mg of sorbic acid. This solution is stable for several days when refrigerated.

4.2 Apparatus

4.2.1 *Steam Distillation Apparatus*

4.2.2 *Volumetric Flask* — 50 ml and 1 litre capacity.

4.2.3 *Pipette* — 5-ml and 20-ml capacity.

4.2.4 *Test Tubes* — 15-ml capacity.

4.2.5 *Spectrophotometer with 10-mm Cells*

4.2.6 *Flask* — 250 ml.

4.3 Procedure

4.3.1 *Preparation of the Sample*

4.3.1.1 *Cheese* — Cut the cheese very finely and mix thoroughly.

4.3.1.2 *Flour confectionery products*

a) *All types of bread and cakes not containing fruits* — Take 1 or half loaf of bread or cake and cut it into slices of 2-3 mm thickness. Spread the slices on the paper and let them dry in a warm place or at room temperature until sufficiently crisp and brittle to grind well. Grind entire sample to pass through 850 micron sieve. Mix well and keep in airtight container.

b) *Bread and cakes containing raisins and fruits* — Take 1 or half loaf of bread or cake and cut it into slices of 2-3 mm thickness. Spread the slices on the paper and let them dry in a warm place or at room temperature until sufficiently crisp. Comminute by passing twice through a food chopper and dry the sample in an oven at 70°C under a pressure of less than 50 mm of mercury.

4.3.2 *Test Portion* — Weigh 1·5 to 2·0 g prepared sample into a distillation tube containing silicon chips. Add 10 ml of 2N sulphuric acid and 10 g magnesium sulphate. Steam, distill the contents maintaining 20-30 ml volume in distillation tube with small burner. Avoid charring. Collect 100-125 ml distillate in 250-ml volume flask within 45 minutes. Rinse condenser with distilled water and dilute the distillate to volume and mix thoroughly.

4.3.3 *Determination* — Pipette 2 ml of test portion and 2 ml of distilled water (for blank) into separate 15 ml test tubes. Add 1 ml of

0·3N sulphuric acid and 1 ml of potassium dichromate solution and heat in a boiling water-bath exactly for five minutes. Immerse tubes in icebath and add 2 ml thiobarbituric acid solution. Replace it in boiling water-bath and boil it for 10 minutes. Cool and determine optical density of solution at 532 nm against blank using matched 1cm cells.

4.3.4 *Plotting of the Calibration Curve* — Pipette 5, 10, 15, 20, 25 ml sorbic acid standard solutions into separate 500 ml volumetric flasks. Dilute each to volume and mix thoroughly and proceed as prescribed in **4.3.3.** Plot the optical density against μg sorbic acid/ml.

4.3.5 *Calculation* — Calculate the sorbic acid content in the sample after reading the corresponding sorbic acid value of the optical density.

$$\text{Percent sorbic acid in the sample} = \frac{125 \times A \times 100}{M}$$

where

A = sorbic acid content obtained from calibration curve, and

M = mass of sample taken.

Percent sodium sorbate = Percent sorbic acid × 1·34

5. SPECTROPHOTOMETRIC METHOD

5.1 Reagents

5.1.1 *Metaphosphoric Acid Solution* — Dissolve 5 g phosphoric acid in 250 ml distilled water and dilute to 1 litre with absolute alcohol.

5.1.2 *Mixed Ethers* — Petroleum ether and anhydrous ether (1 : 1).

5.1.3 *Potassium Permanganate Solution* — Dissolve 15 g potassium permanganate in distilled water, dilute to 100 ml and filter through glass wool.

5.1.4 *Sorbic Acid Solution (Stock)* — Dissolve 200 mg of sorbic acid in 200 ml of mixed ethers.

5.1.4.1 *Working solution* — Dilute 10 ml stock solution to 200 ml with mixed ethers. This solution corresponds to 0·05 mg/ml.

5.1.5 *Reference Solution* — Shake 100 ml mixed ethers with 10 ml metaphosphoric acid solution and dry supernatent ether fraction with 5 g anhydrous granular sodium sulphate.

5.2 Apparatus

5.2.1 *Highspeed Blender*

5.2.2 *Separating Funnels* — 500 ml.

5.2.3 *Volumetric Flasks*

5.2.4 *Graduated Pipettes*

5.2.5 *Ultraviolet Spectrophotometer* — Provided with a 0·5 mm monochromator with silica cells of thickness 20 mm, fitted with ground lids.

5.3 Procedure

5.3.1 *Preparation of the Sample*

5.3.1.1 *Cheese* — Cut the cheese sample very finely and mix thoroughly.

5.3.1.2 *Flour confectionery products* — Proceed as given in **4.3.1.2**(a) and **4.3.1.2**(b).

5.3.2 *Test Portion* — Accurately weigh 10 g prepared sample in the high speed blender cup. Add enough metaphosphoric acid solution to yield a total of 100 ml liquid in mixture. Blend for 1 minute and immediately filter through Whatman No. 3 paper. Transfer 10 ml filtrate to 250-ml separating funnel containing 100 ml mixed ethers and shake for 1 minute. Discard aqueous layer and dry the ether extract with 5 g anhydrous sodium sulphate.

5.3.3 *Determination* — Place the ether solution in a silica cell with a ground lid of thickness 20 mm and measure the absorbance of this solution at 250 nm with respect to the reference solution in a similar silica cell.

5.3.4 *Plotting of the Calibration Curve* — Into a series of four 100 ml volumetric flasks, add 1, 2, 4 and 6 ml of working standard sorbic acid solution and dilute to volume with mixed ethers. Determine absorbance of the solutions at 250 nm. Plot the absorbance against mg of sorbic acid/ml.

5.3.5 *Calculation* — Calculate the sorbic acid content in the sample after reading the corresponding sorbic acid value from the calibration curve.

Percent sorbic acid in the sample = (mg sorbic acid/g sample) × (1/1 000 mg) × 100

$$= \frac{\text{mg sorbic acid}}{10}$$

Percent sodium sorbate = Percent sorbic acid × 1·34

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2